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EVALUATION OF SECTIONING TECHNIQUES FOR ELECTRON MICROSCOPIC ANALYSES OF LOW-DENSITY POLYETHYLENE FOAMS*

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Cell sizes of low-density polyethylene foams in the range of 0.5 to 5 μm are most conveniently measured by SEM analyses. Unfortunately, the cell walls are relatively weak and fragile membranes that either collapse or are severely distorted by conventional surface preparation and sectioning techniques. Sectioning damage can be circumvented to some extent by freeze fracturing. However, fractures tend to propagate through the weakest structural features, they can be associated with severe deformation, even at liquid nitrogen temperatures, and they frequently do not yield planar surfaces for reliable statistical measurements. Therefore, alternate sectioning techniques were evaluated. The most promising techniques are vibrotome sectioning and ultramicrotomy. These techniques are compared with freeze fracturing using SEM examinations of carbon-coated specimens prepared from the same batch of foam.

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Freeze fracturing was accomplished by first partially cutting the specimen with a scalpel, then immersing it in liquid nitrogen until it thermally equilibrates, and finally quickly fracturing it along the partial cut. Figure 1 is typical of structures revealed by freeze fracturing and shows the highly irregular fracture surface produced by this technique.

Both vibrotoming and ultramicrotomy at room temperature produced a skin that completely obliterated the cell structure. Therefore; a Lancer Vibrotome Series 1000 Sectioning System was modified for sectioning at cryogenic temperatures by building a stryofoam insulating chamber around the specimen stage. Platinum-coated razor blades were used as recommended by the manufacturer. Both the speciment and the blade were immersed in liquid nitrogen during cryo-sectioning. Skin formation was reduced somewhat, as shown in Fig. 2, but still did not produce useful structures.

Previous work using cryo-ultramicrotomy on polyethylene foams[1] eliminated skin formation by backfilling with wax. However, most of the common embedding and filling liquids proved to be too viscous to obtain sufficient penetration and filling in our foam. The most satisfactory technique among those tried was to saturate the foams with isopropyl alcohol and to freeze the specimens with the alcohol retained. Isopropyl alcohol is the extraction fluid used in the production of the foams, and it does not alter the structure of the foam. The resultant cell structure shown in Fig. 3 is clearly revealed, although the shearing action of the vibrotome appears to cause some distortion and compression of the cellular structure (indicated by arrows in Fig. 3). This technique does produce relatively flat surfaces.

Ultramicrotomy was performed with a Sorvall MT-6000 cryo-ultramicrotome equipped with an FS-1000 cryosectioning system. Most of the specimens were sectioned at -160 C, which is well below the glass transition temperature for polyethylene[2]. Standard glass knifes were prepared by fracturing to form a 45 degree cutting edge. The excellent delineation of the cell structure obtained with cryo-ultramicrotomy is shown in Fig. 4. Only minimal plastic smearing occurred, and the sectioned surface was relatively flat. The success of both techniques was predicated on the use of small specimen sizes of the order of 1-mm square.

Satisfactory sectioning of low-density polyethylene foam can be accomplished at cryogenic temperatures if the foam is back filled with isopropyl alcohol. Both cryo-vibrotoming and cryo-ultramicrotomy yield essentially identical structures, although remnant sectioning damage is slightly heavier after vibrotoming than after ultramicrotomy. In addition, comparison of Figs. 1, 3, and 4 shows that the cell size revealed by cryo-vibrotoming and cryo-ultramicrotomy is significantly larger than that revealed by freeze fracturing, and this definitely indicates that structures revealed by freeze fracturing are not representative of the general internal structure.[3]

1. E. F. Koch, Ann. Proc. EMSA 26(1968)412.
2. Z. Tadmor and C. D. Gogos, Principles of Polymer Processing, New York: John Wiley and Sons (1979)38.
3. This work was performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract #W-7405-ENG-48; the ultramicrotomy was performed under the guidance of C. N. Schooley and D. B. Davis, University of California, Berkeley.

Fig. 1. Freeze fracture.

Fig. 2. Cryo-vibrotome, unfilled

Fig. 3. Cryo-vibrotome, filled.

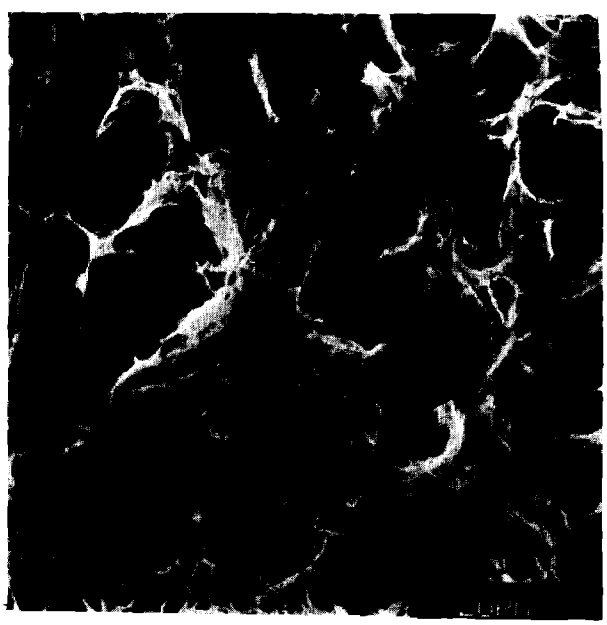

Fig. 4. Cryo-ultramicrotome, filled.